

Small Column Ion Exchange
Testing of SuperLig® 644 for
Removal of ^{137}Cs from Hanford
Waste Tank 241-AP-101 Diluted
Feed (Envelope A)

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September 2002

Prepared for Bechtel National Inc.
under Contract No. 24590-101-TSA-W0000-0004

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Battelle Pacific Northwest Division
Richland, Washington 99352

Completeness of Testing

This report describes the results of work and testing specified by TSP-W375-00-00028, Rev. 1 and CHG-TP-41500-013, Rev. 0 and TP-RPP-WTP-013, Rev. 0. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

Gordon H. Beeman, Manager
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Date

Summary

The River Protection Project-Waste Treatment Plant (RPP-WTP) baseline process for ^{137}Cs removal from Hanford high-level tank waste is ion exchange. The current flowsheet includes the use of Cs-selective, organic ion exchanger SuperLig[®] 644 (SL-644) material for Cs removal from the aqueous waste fraction. This material has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT. The RPP-WTP Development Requirements Document (DRD)¹ task 8.2.6 and the RPP-WTP Research and Technology schedule² identify Cs and Tc ion exchange process verification tests [WTP Request for Proposal reference Standard 2 item (a)(3)(ii)].

Battelle Pacific Northwest Division (PNWD) was contracted to perform Cs ion exchange studies under contract 24590-101-TSA-W000-0004, and work breakdown structure BN.02.08.05. The Cs ion exchange activities are further defined in Technical Scoping Statement B-42a, which is included in Appendix C of the *Research and Technology Plan*². These studies are to verify design and operating parameters for plant-scale ion exchange systems. Test results will also be used to validate ion exchange models.

Objectives

The Cs ion exchange test objectives were to develop load and elution breakthrough profiles using AP-101 diluted feed (AP-101DF); produce and characterize the Cs eluate for use in evaporation tests; remove ^{137}Cs from the AP-101DF to meet LAW vitrification criteria; and develop batch distribution coefficient values for AP-101DF. The final effluent was to contain $<1.75\text{E-}5$ Ci ^{137}Cs per mole Na, equivalent to <0.087 $\mu\text{Ci }^{137}\text{Cs}/\text{mL}$, based on a 20 wt% waste Na_2O loading in the waste glass. Direction from BNI for calculating Na_2O loading was later reduced to 14 wt%. All testing objectives were met.

Conduct of Test

This report summarizes testing of the SL-644 in batch-contact studies and in a dual small-column system. The test matrix was Hanford tank waste 241-AP-101 (Envelope A) diluted to 5 M Na^+ (AP-101DF). Batch contacts were performed with the waste at three Cs concentrations with a phase ratio of 100 (liquid volume to exchanger mass ratio) with both SL-644 and crystalline silicotitanate (IE-911, an alternative Cs ion exchanger). Ion exchange processing was conducted with two small columns in series with resin bed volumes (BVs) of 10.6 mL ($L/D = 4.4$) during the conditioning phase with 0.25 M NaOH, and 9.7 mL ($L/D = 4$) during the AP-101DF loading phase. Proper functioning of the ion exchange apparatus and resin beds had initially been tested with an AW-101 simulant. Approximately 1.2 L, equivalent to 112 BVs of AP-101DF waste were processed. All ion exchange process steps were tested

¹ PL-W375-TE00002, Rev. 1, River Protection Project Waste Treatment Plant Development Requirements Document, October 31, 2000, M. E., Johnson and T. W. Crawford, CH2MHill Hanford Group, Inc., Richland, WA. DRAFT.

² Barnes S, R Roosa, and R Peterson. 2002. *Research and Technology Plan*. 24590-WTP-PL-RT-01-002 Rev. 1, RPP-WTP project.

including resin bed preparation, loading, feed displacement, water rinse, elution, eluant rinse, and resin regeneration.

Results and Performance Against Objectives

The batch-contact performance data are summarized in Table S1. The Cs λ value (column distribution ratio) represents a measure of the effective capacity of the SL-644 resin; the higher the λ value, the higher loading capacity. Results from the SL-644 batch-contact tests, resulted in an estimated batch distribution (K_d) value of 690 mL/g in the feed condition (Na/Cs mole ratio of 1.4 E+5). Using a bed density of 0.230 g/mL in the 0.25 M NaOH condition, the Cs λ value is predicted to be 160 BVs. The IE-911 feed condition K_d value was interpolated to be 710 mL/g. With a bed density of 1.04 g/mL, the estimated λ value is 740 BVs.

Table S1. Summary of Performance Measures

Flow rate (BV/h)	Extrapolated 50% Cs breakthrough, BV		Composite DF ⁽¹⁾	Maximum DF ⁽²⁾	K _d , mL/g (feed condition)		Predicted Cs λ , BV	
	Lead column	Lag column			SL-644	IE-911	SL-644	IE-911
3	190	—	7.78E+5	1.4E+6	690	710	160	740
(1) The decontamination factor was calculated by dividing the feed Cs concentration by the composite effluent Cs concentration. (2) The maximum DF was obtained by dividing the feed Cs concentration by the lowest lag column sample Cs concentration.								

The ion exchange column processing performance data are also summarized in Table S1. The maximum ion exchange Cs breakthrough obtained was 0.27% at 112 BVs (regeneration condition). Based on the AP-101DF column load data, the lead column 50% Cs breakthrough was extrapolated to be 190 BVs (resin BV in the 0.25 M NaOH regeneration condition). A 50% Cs breakthrough value for the lag column could not be estimated; virtually no measurable Cs broke through the lag column. The decontamination factor (DF) for ¹³⁷Cs, listed in Table S1, was based on ¹³⁷Cs concentration in the feed divided by the ¹³⁷Cs concentration in the composite effluent. The DF for ¹³⁷Cs was 7.78 E+5, and is equivalent to an effluent with a ¹³⁷Cs concentration of 1.1E-4 μ Ci/mL. The maximum DF, 1.4 E+6, measured the best performance that could be expected from this column system.

The LAW vitrified waste form must be no greater than 0.3 Ci ¹³⁷Cs/m³. This limit can be converted to ¹³⁷Cs concentration in the Cs ion exchange effluent of 0.125 μ Ci ¹³⁷Cs/mL.³ The product effluent ¹³⁷Cs concentration was a factor of 1000 below this contract limit.

³ The conversion requires the following assumptions: Envelope A LAW will contain 14 wt% Na₂O, all Na comes from the tank waste, the glass density is 2.66 g/mL, and the waste Na concentration is 5 M Na. For the maximum 20 wt% waste Na₂O loading, the Cs ion exchange effluent must be less than 0.087 μ Ci ¹³⁷Cs/mL.

The lead column was eluted with 0.5 M HNO₃ to C/C₀ of 1% in 12 BVs with >94% of the ¹³⁷Cs contained in 2.5 BVs of eluant. The peak ¹³⁷Cs C/C₀ value was 64 (based on 1-BV collection increments of nominally 10-mL).

Quality Requirements

This work was designated as QL-3 per the RPP-WTP Quality Assurance Program, BNFL-5193-QAP-01, Rev. 6. PNWD implemented the RPP-WTP quality requirements by performing work in accordance with the quality assurance plan, CHG-QAPjP, Rev. 0.

Issue

U and Pu were found in the Cs eluate, indicating these materials exchanged onto the resin. No additional issues were observed.

Terms and Abbreviations

AV	apparatus volume
BV	bed volume
C/C ₀	analyte concentration in column effluent divided by analyte concentration in feed
CMC	Chemical Measurement Center
DI	deionized
EQL	estimated quantitation level
F-factor	mass of dry ion exchanger divided by mass of wet exchanger
FMI	Fluid Metering, Inc., Syosset, NY
GEA	gamma energy analysis
HLRF	High Level Radiation Facility
IBC	IBC Advanced Technologies, Inc., American Fork, Utah
IC	ion chromatography
ICP-AES	inductively coupled plasma/atomic emission spectrometry
ICP-MS	inductively coupled plasma/mass spectrometry
λ	column distribution ratio
L/D	length over diameter ratio
M	molarity, moles/liter
MDL	method detection limit
MRQ	minimum reportable quantity
NMRQ	no minimum reportable quantity
NPT	National Pipe Thread
PSD	particle size distribution
SAL	Shielded Analytical Laboratory
TIC	total inorganic carbon
TOC	total organic carbon

Contents

Summary	iii
Terms and Abbreviations	vii
1.0 Introduction	1.1
2.0 Experimental	2.1
2.1 SL-644 Resin	2.1
2.2 Crystalline Silicotitanate	2.2
2.3 AP-101 Diluted Feed	2.3
2.4 Batch Contacts	2.5
2.5 Column Run Experimental Conditions	2.6
2.5.1 Ion Exchange Column System	2.6
2.5.2 Resin Bed Preparation	2.7
2.5.3 Ion Exchange Column Test with AP-101DF	2.9
2.5.4 Sample Analysis	2.11
3.0 Results and Discussion	3.1
3.1 Batch Contact Results	3.1
3.2 Column Test	3.3
3.2.1 Initial Resin Conditioning	3.3
3.2.2 Loading	3.3
3.2.3 Feed Displacement and Rinse	3.6
3.2.4 Elution and Eluant Rinse	3.6
3.2.5 Regeneration	3.13
3.2.6 Activity Balance for ¹³⁷ Cs	3.13
3.2.7 SL-644 Resin Volume Changes	3.14
4.0 Conclusions	4.1
5.0 References	5.1
Appendix A	A.1
Appendix B	B.1
Appendix C	C.1
Appendix D	D.1
Appendix E	E.1
Appendix F	F.1

Figures

Figure 3.1. ^{137}Cs Distribution Coefficients (K_d) for AP-101DF (T = 26°C)	3.1
Figure 3.2. Estimated ^{137}Cs λ Values for AP-101DF (T = 26°C).....	3.3
Figure 3.3. ^{137}Cs Breakthrough Curves for AP-101DF Sample, Probability Plot.....	3.5
Figure 3.4. ^{137}Cs Elution and Eluant Rinse of the Lead Column.....	3.8
Figure 3.5. (a and b) Comparison of Bed Volumes of the Lead and Lag Columns	3.16

Tables

Table S1. Summary of Performance Measures.....	iv
Table 2.1. Dry Particle-Size Weight-Percent Distribution of Various Batches of As-Received SL-644.....	2.1
Table 2.2. SL-644 Properties	2.2
Table 2.3. Composition of AP-101DF (Envelope A)	2.4
Table 2.4. Initial Cs Concentrations in the AP-101DF Solutions Used for the Batch Distribution Tests.....	2.5
Table 2.5. Bed Conditioning and Simulant Shakedown Testing Parameters	2.9
Table 2.6. Experimental Conditions for AP-101DF Ion Exchange	2.10
Table 2.7. Sampling Interval and Analyses	2.11
Table 3.1. Dry Bed Density	3.2
Table 3.2. Decontamination Factors for ^{137}Cs from AP-101DF	3.4
Table 3.3. AP-101DF Cs-Decontaminated Product Effluent.....	3.7
Table 3.4. Inorganic and Organic Analytes in the Lead Column Eluate Composite.....	3.9
Table 3.5. Radionuclides in the Lead Column Eluate Composite ⁽¹⁾	3.11
Table 3.6. Composition of Regeneration Solution.....	3.13
Table 3.7. Activity Balance for ^{137}Cs	3.13
Table 3.8. SL-644 Bed Volume Changes	3.15
Table B.1. Sample Identification	B.1

1.0 Introduction

The U. S. Department of Energy plans to vitrify tank wastes at the Hanford Site in preparation for permanent disposal. Before vitrification, tank wastes will be divided into low-activity and high-activity fractions through specific pretreatment processes. The pretreatment flow sheet for the Hanford high-level tank wastes includes the use of SuperLig[®] 644 (SL-644) material for ¹³⁷Cs removal from the aqueous waste fraction. The SL-644 is a Cs-selective, organic, ion exchanger and has been shown to be effective in removing Cs from a variety of Hanford tank wastes (Hassan, McCabe, and King 2000; Hassan, McCabe, King and Crowder, 2000; Hassan, King, McCabe, and Crowder 2001; King, Hassan, and McCabe 2001; Kurath, Blanchard, and Bontha 2000a; Kurath, Blanchard, and Bontha 2000b). The SL-644 has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT.

This report summarizes batch-contact studies of SL-644 and crystalline silicotitanate⁴ (IE-911, UOP LLC, Des Plaines, IL), and dual small-column testing of the SL-644 ion exchange material. The test matrix was 241-AP-101 Hanford tank waste diluted to 5 M Na⁺ (hereafter referred to as AP-101DF) (Goheen, et al., 2001). Approximately 1.2 L of AP-101DF were processed through the ion exchange column system. The ion exchange process steps tested include resin bed preparation, loading, feed displacement, water rinse, elution, and resin regeneration.

The objectives of this work were to:

- determine batch distribution coefficients as a function of Na/Cs molar concentration for SL-644 and IE-911 in the AP-101DF matrix.
- demonstrate the ¹³⁷Cs decontamination of Envelope A tank waste sample AP-101DF using SL-644 columns and provide a Cs-decontaminated sample for downstream process testing (i.e., ⁹⁹Tc removal, low activity waste (LAW) melter feed testing, and LAW vitrification).
- develop loading and elution breakthrough profiles for SL-644 columns.
- produce and characterize the Cs eluate solution from SL-644 columns for use in evaporation tests and high-level waste (HLW) vitrification.
- demonstrate the effectiveness of all SL-644 ion exchange process steps including loading, feed displacement, deionized (DI) water washing, elution and resin regeneration.⁵

⁴IE-911 is an alternative Cs ion exchanger and is being tested in parallel to SL-644 only with batch contacts. The IE-911 was developed and supplied by UOP LLC, Des Plaines, IL.

⁵ Test Specification: "Tank 241-AN-102 and 241-AP-101 Ion Exchange," TSP-W375-00-00028, Rev. 1, M. E. Johnson, CH2M Hill Hanford Group, Dec. 11, 2000 and Test Plan "Actual Waste Ion Exchange Testing for the RPP-WTP Project," CHG-TP-41500-013, Rev. 0, D. L. Blanchard, Jan. 24, 2000.

2.0 Experimental

2.1 SL-644 Resin

Initial AP-101DF batch contacts were conducted with SL-644 resin batch 981112YK-N3-16/18. This material batch had a distinct salt and pepper appearance with a significant light gray fraction. Initial batch distribution studies with this SL-644 batch on AW-101 simulant indicated it performed well and it was used for AP-101DF batch distribution testing. Unfortunately, later simulant column testing proved this material inadequate (Fiskum, Blanchard, and Arm 2002). Additional SL-644 was obtained from IBC production batch number 010319SMC-IV-73. This material batch exhibited a black-red appearance peppered with light brown specs.

Before sampling SL-644 from the IBC-supplied plastic bottle, the bulk resin bottle was turned several times to produce a well-mixed material. Sub-samples were removed immediately after mixing. The sieve results of the two resin batches were previously reported (Fiskum, Blanchard, and Arm 2002) and are reproduced in Table 2.1. The 212- to 425- μm particle size dry-sieved fraction was used in the ion exchange columns.⁶ The average particle size corresponded to 540- μm in diameter, expanded in 3 M NaOH-2 M NaNO₃-0.1 M KNO₃ (Fiskum, Blanchard, and Arm 2002). As a general rule, the column diameter should be 20 times greater than the resin particle diameter to minimize wall effects (Korkisch). Given the diameter of the column at 1.46 cm, the column diameter was 27 times the average diameter of the 212- to 425- μm diameter dry-sieved resin particles expanded in caustic solution.

Table 2.1. Dry Particle-Size Weight-Percent Distribution of Various Batches of As-Received SL-644

Sieve Size ⁽¹⁾	Particle Size (μm)	981112YK-N3-16/18 wt %	010319SMC-IV-73 wt %
18	>1000	1.78	0.06
30	600-1000	55.25	37.27
40	425-600	20.12	38.23
50	300-425	13.76	18.01
70	212-300	7.93	6.08
100	150-212	1.11	0.26
140	106 -150	0.02	0.06
>140	<106	0.03	0.03

(1) U. S. standard sieve size corresponds to ASTM E-11 specification.

Properties of the 981112YK-N3-16/18 and 212- to 425- μm 010319SMC-IV-73 SL-644 resins have been previously reported (Fiskum, Blanchard, Arm 2002) and selected properties are reproduced in Table 2.2. The F-factor is the ratio of the dry mass of exchanger to the initial mass of the exchanger, and was

⁶ This particle size distribution was used successfully in AW-101 simulant testing.

determined at the same time the batch contact samples and column resin fractions were weighed. The F-factor was obtained by drying approximately 0.5 g resin, under vacuum, at 50°C to constant mass. The F-factor was determined on the H-form (hydrogen form or acid form) and the as-received form of the resin. The F-factor for the Na-form resin was performed differently because of stability problems observed in prior tests on this resin form (Steimke et al. 2001). Drying to constant mass under vacuum at ambient temperature was considered adequate for removing water from the Na-form resin.⁷ The L-factor represents the fractional mass remaining after washing the as-received resin form with 0.5 M HNO₃ and DI water, correcting for residual water content as described above. The I_{Na} represents the fractional mass gain upon conversion from the H-form to the Na-form, correcting for water content as described above.

Table 2.2. SL-644 Properties

Property	981112YK-N3-16/18	010319SMC-IV-73
Sieve fraction	As-received	212- to 425- μ m
Bulk density, g/mL ⁽¹⁾	0.80	0.74
F-factor, as-received	0.891	0.8773
L-factor, conversion to H-form, fractional mass remaining	0.493	0.538
F-factor, H-form	0.939	0.932
I _{Na} , fractional mass gain from H-form to Na-form	1.29	1.25
(1) Bulk density based on resin as-supplied by IBC, no drying, washing, or conditioning.		

2.2 Crystalline Silicotitanate

Crystalline silicotitanate IE-911, Lot 2081000009, was obtained from UOP LLC, Des Plaines, IL. The UOP particle size specification of this material was 30-60 mesh. The material was provided in the hydrogen form. A 5-g aliquot was converted to the sodium form by soaking in 40 mL 1 M NaOH for two days. The NaOH solution was then decanted and the IE-911 was rinsed with five successive volumes of DI water. The resin was then dried at 95°C overnight. The F-factor, 0.9630 was determined at the same time the batch contact samples were weighed. This was obtained by drying approximately 0.5 g at 95°C to constant mass.

⁷ After initial drying at ambient temperature under vacuum to constant mass, the resin was heated to 50°C. The heated product appeared (visual inspection) to have degraded thus potentially nullifying subsequent mass measurements.

2.3 AP-101 Diluted Feed

Samples of tank 241-AP-101 (Envelope A) waste were received in the High Level Radiation Facility (HLRF) in 2000. The homogenization, dilution, subsampling, and analysis of this material have been previously described (Goheen et al. 2002). The total volume of AP-101DF available for Cs ion exchange processing was about 1.2 L. The feed composition is summarized in Table 2.3.

Table 2.3. Composition of AP-101DF (Envelope A)

Cations, M	
Na ⁺	4.97 E+0
K ⁺	7.61 E-1
Cs ⁺	3.68 E-5 ⁽¹⁾
Uranyl	1.7 E-4
Mole Ratios	
Na/Cs mole ratio	1.4 E+5
K/Cs mole ratio	2.1 E+4
Anions, M	
AlO ₂ ^{- (2)}	2.59 E-1
Cl ⁻	4.1 E-2
F ⁻	< 1.2 E-1 ⁽³⁾
CO ₃ ²⁻	4.46 E-1
CrO ₄ ^{-2 (2)}	2.92 E-3
NO ₂ ⁻	7.1 E-1
NO ₃ ⁻	1.68 E+0
OH ⁻	1.94 E+0
PO ₄ ^{-3 (2)}	1.2 E-2
SO ₄ ⁻²	4.8 E-2
Oxalate	< 1 E-2
TOC, g/L	1.64 g/L
Radionuclides, (μCi/mL)	
Total alpha	2.6 E-04
⁶⁰ Co	2.5 E-03
⁹⁰ Sr	7.1 E-02
⁹⁹ Tc	3.93 E-2
⁹⁹ Tc as pertechnetate	3.46 E-2
¹³⁷ Cs	1.26 E+2
¹⁵⁴ Eu	< 7 E-4
²³⁹⁺²⁴⁰ Pu	1.10E-4
²⁴³⁺²⁴⁴ Cm	2.12E-6
Solution density, g/mL	1.256
<p>⁽¹⁾ Total Cs reported by Goheen et al. 2002 may be biased low. Reported value does not agree with ¹³⁷Cs determination by GEA and application of isotopic distribution (¹³³Cs 60.6 wt%, ¹³⁵Cs 15.4 wt%, ¹³⁷Cs 24.0 wt%) where a total Cs concentration of 6.04 μg/mL or 4.5 E-5 M is calculated.</p> <p>⁽²⁾ Al, Cr, and P determined by ICP-AES. The anionic form is assumed on the basis of waste chemistry.</p> <p>⁽³⁾ F concentration is an upper bound; co-eluting anions positively interfered with peak integration.</p>	

2.4 Batch Contacts

The batch contacts were performed with the H-form of SL-644 batch 981112YK-N3-16/18. The Na-form of IE-911 was tested in parallel with the SL-644. Initial batch contacts were performed using AP-101DF at three different Cs concentrations. Aliquots of the AP-101DF subsamples were tested without spiking, additional aliquots were spiked with 0.1 M CsNO₃ to obtain stock solutions of nominally 1E-3 M and 5 E-3 M Cs. The initial Cs concentrations in the AP-101DF stock contact solutions and the corresponding Na/Cs and K/Cs mole ratios are given in Table 2.4. After discerning the SL-644 batch 981112YK-N3-16/18 was insufficient to remove Cs under column loading conditions (Fiskum, Blanchard, and Arm 2002), another batch contact with AP-101DF was performed using the H-form of SL-644 batch 010319SMC-IV-73, 212- to 425- μ m dry particle size. The scope of this second test was much reduced in order to conserve AP-101DF feed for use in subsequent column ion exchange and vitrification tests. Thus only the mid-range Cs concentration was tested in duplicate.

Table 2.4. Initial Cs Concentrations in the AP-101DF Solutions Used for the Batch Distribution Tests

Solution	Target Initial Cs Conc. [M]	Target Nominal Na/Cs⁽¹⁾ mole ratio	Target Nominal K/Cs⁽¹⁾ mole ratio
SL-644 Batch 981112YK-N3-16/18 and IE-911			
Un-spiked	3.68E-5	1.4 E+5	2.1 E+4
Cs Spike 1	1.0 E-3	5.0 E+3	7.6 E+2
Cs Spike 2	5.0 E-3	1.0 E+3	1.5 E+2
SL-644 Batch 010319SMC-IV-73			
Cs Spike 3	1.0 E-3	5.0 E+3	7.6 E+2
(1) Na ⁺ and K ⁺ are the primary cations that compete with Cs ⁺ for ion exchange with SL-644 and IE-911.			

The batch-contact tests were performed in duplicate at a phase ratio of approximately 100 mL/g (liquid volume to exchanger mass). Typically, 0.1 g of SL-644 exchanger was contacted with 10 mL of AP-101DF, and 0.05 g of IE-911 and SL-644 for Cs Spike 3 were contacted with 5 mL AP-101DF. The exchanger mass was determined to an accuracy of 0.0002 g. The waste volume was transferred by pipet and the actual volume was determined by mass difference with an accuracy of 0.0002 g and the solution density. Samples were agitated in an orbital shaker for approximately 24 hours for SL-644 and 72 hours for IE-911. The temperature was not controlled but was generally constant at 26 \pm 1°C during the 3 days of contact.

All batch-distribution measurements were determined by measuring ¹³⁷Cs on both the stock solution (initial concentration) and the contacted solution (final concentration). The initial Na and K concentrations were determined with inductively coupled plasma-atomic emission spectrometry (ICP-AES). Initial ¹³³Cs concentrations were confirmed by inductively coupled plasma-mass spectrometry (ICP-MS).

The batch distribution coefficient, K_d (with units of mL/g), was determined using the following relationship:

$$K_d = \frac{(C_0 - C_1)}{C_1} * \frac{V}{M * F} \quad (1)$$

where C_0 = initial ^{137}Cs concentration
 C_1 = final ^{137}Cs concentration
 V = volume of the liquid sample (mL)
 M = mass of the ion exchanger (g) (SL-644 H-form mass corrected for the Na-form mass increase or Na-form IE-911 mass)
 F = dried resin mass divided by the initial resin mass.

The dry bed resin density, ρ , was obtained according to the following equation:

$$\rho = \frac{m * L * F * I_{Na}}{BV} \quad (2)$$

where ρ = dry Na-form g of resin per mL of resin
 m = resin mass in the column, as-received form
 L = fractional mass remaining after washing (0.538)
 F = water loss factor, as-received form (0.877)
 I_{Na} = fractional mass gain on conversion from H-form to Na-form (1.25) (this factor is set to 1 when calculating the dry bed density in the H-form or 0.5 M HNO_3 feed)
 BV = resin bed volume in the feed (discussed in section 3.2.7).

The Cs λ value (column distribution ratio) is a function of the feed condition batch distribution coefficient and the dry bed density, and is obtained as shown in Equation 3.

$$\lambda = K_d * \rho \quad (3)$$

2.5 Column Run Experimental Conditions

2.5.1 Ion Exchange Column System

A schematic of the ion exchange column system is shown in Figure 2.1. The system consisted of two small columns containing the ion exchange material, a small metering pump, three valves, a pressure gauge, and a pressure relief valve. Valves 1, 2, and 3 were three-way valves that could be turned to the flow position, sample position, or no-flow position. Valve 1 was placed at the outlet of the pump and was used to eliminate air from the system, purge the initial volume of the system, or isolate the columns from the pump. Valves 2 and 3 were primarily used for obtaining samples and could also be used to isolate the

columns from the rest of the system. The columns were connected in series with the first column referred to as the lead column and the second column referred to as the lag column.

The columns were prepared at Savannah River Technology Center Glassblowing Laboratory. Each column consisted of a 15-cm glass column with a 24/40 taper ground-glass fitting on top and a threaded fitting on the bottom. A polyethylene bushing was installed in each glass-threaded fitting to accommodate a ¼-in stainless steel National Pipe Thread (NPT) fitting. The inside diameter of each column was 1.46 cm, which corresponded to a volume of 1.67 mL/cm. A stainless steel, 200-mesh screen supported the resin bed. A decal millimeter scale affixed to the column allowed for measurement of resin bed height and thus shrinkage and swelling. The upper section contained four entry ports and a taper joint with screw cap that securely fitted the column. The lead column assembly used a pressure relief valve (10 psi trigger), pressure gauge, and sample inlet; the remaining port was plugged. The lag column assembly used one port for sample entry, and the other three ports were plugged. In both columns, the inlet sample lines extended through the port opening to the top of the column. The connecting tubing was 1/8-in OD, 1/16-in ID polyethylene. Valved quick-disconnects (Cole Parmer, Vernon Hills, IL) were installed in-line to allow for ease of column switching. A FMI QVG50 pump (Fluid Metering, Inc., Syosset, New York) equipped with a ceramic and Kynar[®] coated low-flow piston pump head was used for all fluid introduction. The flow rate was controlled with a remotely operated FMI stroke-rate controller. The pump was calibrated with the stroke-rate controller and could provide pumping rates from 0.08 to 16 mL/min. The volume actually pumped was determined using the mass of the fluid and the fluid density. The pressure indicated on the pressure gauge remained below 5 psi during all runs. The total holdup volume of the Cs ion exchange system was the summed volume of all fluid-filled parts and was estimated to be 42 mL.

2.5.2 Resin Bed Preparation

The initial SL-644 resin and bed preparations were performed using a procedure similar to that recommended by Hassan et al. (1999). Before packing the resin beds, aliquots of SL-644 resin were placed in separate beakers. The two resin aliquots (4.17 and 4.20 g lead and lag columns, respectively) were soaked in about 40 mL of 1 M NaOH (~10:1 solution to resin volume ratio) with light agitation for 2 h. The NaOH was decanted, and the SL-644 was slurried with about 40 mL of DI water. The resin was then quantitatively transferred into the columns using additional DI water to aid the transfer. Both of the resin beds were individually washed with 0.5 M HNO₃. After resin washing, the corrected sodium form SL-644 mass was calculated to be 2.46 g and 2.48 g (dried at ambient temperature under vacuum) in the lead and lag columns, respectively, according to the following equation:

$$M_{bed} = M_{AR} * F_{AR} * L * I_{Na} \quad (4)$$

where M_{bed} = resin bed mass

M_{AR} = resin mass loaded in the column, as-received form, 4.17 g lead and 4.20 g lag columns

F_{AR} = F-factor for the as-received resin, 0.877

L = fractional mass remaining after conversion to the H-form, 0.538

I_{Na} = fractional mass gain on conversion from the H-form to the Na-form, 1.25.

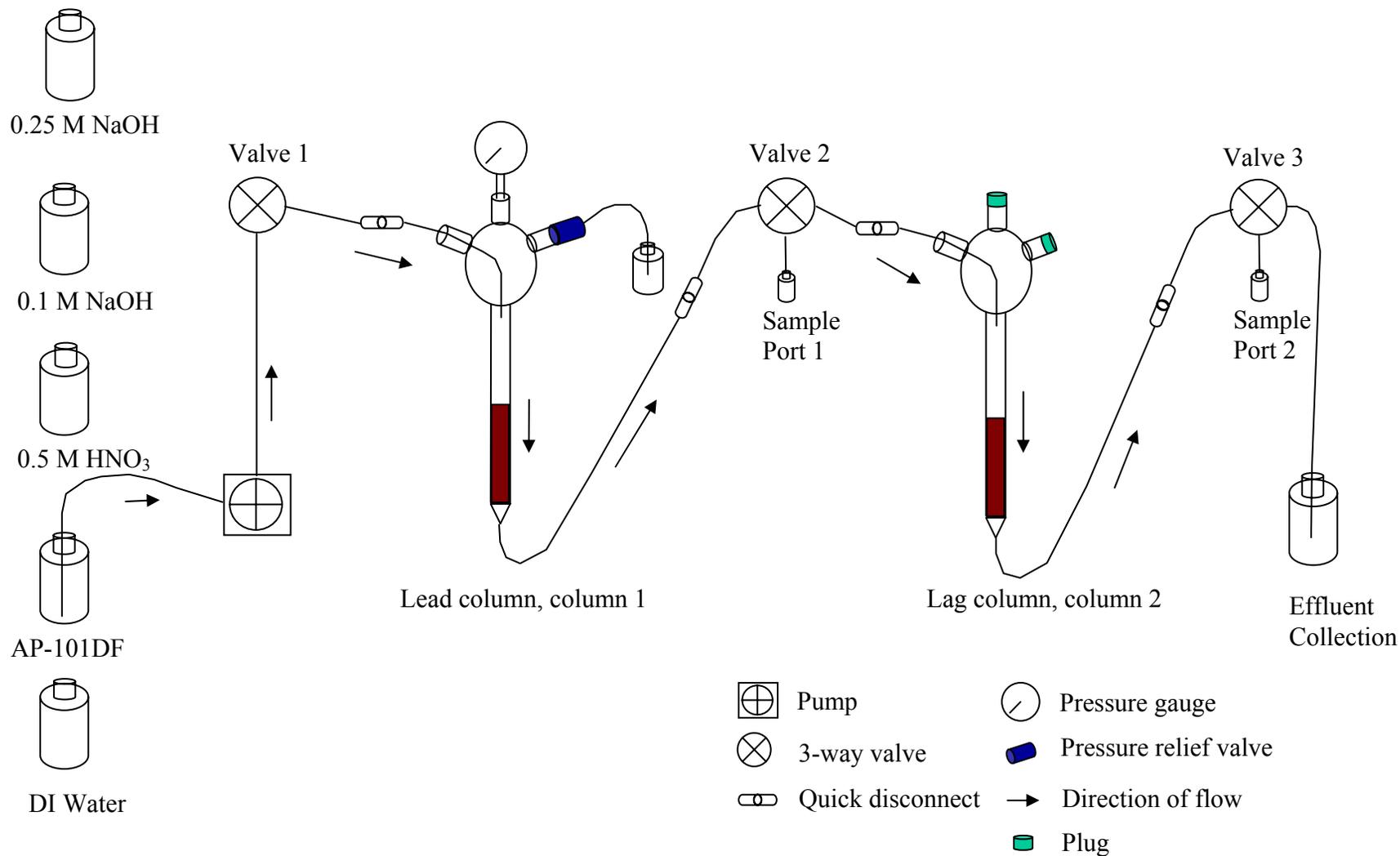


Figure 2.1. Cesium Ion Exchange Column System

The entire ion exchange system was rinsed with DI water, regenerated with 0.25 M NaOH, and then used for a full shakedown experiment with AW-101 simulant (Fiskum, Blanchard, and Arm 2002). Both columns were individually eluted and rinsed. A summary of the shakedown test operations is provided in Table 2.5.

Table 2.5. Bed Conditioning and Simulant Shakedown Testing Parameters

Process Step	Solution	Average Volume			Flow Rate, BV/h
		BV ⁽¹⁾	AV ⁽²⁾	mL	
Pre-Conditioning, Lead and Lag Columns Separate					
Wash	0.5 M HNO ₃	13.5		150	0.87
DI water rinse	DI water		3.0	67	2.1
Regeneration	0.25 M NaOH	5.1		56	2.0
Loading, Feed Displacement, and Rinse, Columns in Series					
Regeneration (cont.)	0.25 M NaOH	3.7		41	1.9
Load	AW-101 simulant	143		1576	2.6
Feed Displacement	0.1 M NaOH		2.9	121	2.5
Rinse	DI water		2.0	84	2.5
Elution, Lead and Lag Columns Separate					
Elution	0.5 M HNO ₃	12		132	0.92
Eluant rinse	DI water		2.7	60	2.6
(1) BV = bed volume (11 mL in 0.25 M NaOH regeneration condition)					
(2) AV = apparatus volume (42 mL for columns in series; 23 mL for lead column processing and 21 mL for lag column processing)					

2.5.3 Ion Exchange Column Test with AP-101DF

The beds had been stored approximately 3 weeks in the H-form in DI water since the end of the simulant AW-101 column run. The resin beds were fluidized with DI water. Then the ion exchange system was loaded into the Shielded Analytical Laboratory (SAL) hot cell with the resin in the H-form. All subsequent processing was performed in the hot cells at temperatures ranging from 25 to 29°C. The entire system was initially cycled in the hot cell with 0.25 M NaOH, DI water, 0.5 M HNO₃, and DI water sequentially through the lead and lag columns. The bed conditioning, AP-101DF loading,⁸ feed displacement, and DI water rinse steps were conducted by passing these solutions through both resin beds connected in series. The elution, elution rinse, and the regeneration steps were conducted on the lead column only. The experimental conditions for each process step are shown in Table 2.6. The resin bed volume (BV) is the volume in 0.25 M NaOH. For the AP-101DF test, the BV was measured at 10.6 mL in the regeneration condition just prior to waste loading. The AP-101DF effluent was collected in four effluent bottles. The first bottle collected 64 mL, slightly over one apparatus volume (AV), and consisted primarily of the displaced regeneration solution. The following effluent collections captured nominally

⁸ AP-101DF processing represents the second Cs load and elute cycle for the resin beds.

350-mL AP-101DF fractions. After ascertaining ^{137}Cs activity was low, the three 350-mL fractions were combined into one composite for follow-on Tc ion exchange.

The solution above the lead resin bed was drained to within about 1 cm of the resin surface when switching to the next process step. Then nominally 5-mL of the solution were allowed to build up above the resin bed prior to commencement of flow. This helped minimize reagent mixing thus speeding the reagent transition through the column system.

Table 2.6. Experimental Conditions for AP-101DF Ion Exchange

Process step	Solution	Total Volume			Flow rate		Time, h	T, °C
		BV ⁽¹⁾	AV ⁽²⁾	mL	BV/h	mL/min		
Bed conditioning	0.25 M NaOH	9.8	2.5	104	1.9	0.33	5.2	25
Bed conditioning	DI water	5.7	1.4	60.6	2.7	0.48	2.1	25
Bed conditioning	0.5 M HNO ₃	7.8	2	82.2	2.5	0.44	3.1	25
Bed conditioning	DI water	6.9	1.8	74	2.8	0.49	2.5	25
Regeneration	0.25 M NaOH	9.6	2.4	103	1	0.18	9.6	25
Loading lead column	AP-101 Feed	112.2	-	1189	2.8	0.50	37.8	26-28
Loading lag column ⁽³⁾	AP-101 Feed	109.4	-	1159	2.8	0.50	37.8	26-28
Feed displacement	0.1 M NaOH	13.2	3.3	140	2.74	0.48	5.1	28
DI water rinse	DI water	8.7	2.2	92	2.9	0.51	3.2	28
Elution of lead column								
Elution	0.5 M HNO ₃	19.3	-	204	0.88	0.16	24.6	28-29
Eluant rinse	DI water	6.9	3.19	73.7	2.9	0.51	4.5	28-29
Regeneration	0.25 M NaOH	4.7	2.17	50	0.9 - 1.5	0.16 - 0.27	6.2	23
Rinse	DI water	5	2.31	53.1	2.59	0.46	1.9	24
(1) BV in 0.25 M NaOH regeneration condition (10.6 mL),								
(2) AV (42 mL for sequential column format, 23 mL for lead column only)								
(3) The feed volume through the lag column is reduced because of sampling from the lead column.								

The sampling and analysis protocol is shown in Table 2.7. During the loading phase, small samples (about 2 mL) were collected from the lead column starting at 7 BVs, then at 15 BVs and continuing in nominal 10-BV increments of feed from both the lead and lag columns. The flow rate ranged from 2.99 to 3.23 BV/h in the feed condition, which was equivalent to 2.74 to 2.96 BV/h when the resin bed was expanded in the regeneration solution. Flow rate doubled during sample collection from the lead column because of the strong siphoning effect at the sample port. Thus, for the 13 samples collected from the lead column, and for 2 minutes each sample, AP-101DF fed at a nominal flow rate of 6 BV/h. The feed displacement, DI water rinse, elution, and elution rinse samples were taken at 1-BV increments at flow rates shown in Table 2.6.

Table 2.7. Sampling Interval and Analyses

Process Step	Lead Column	Lag Column	Approximate Sample Size (mL)	Analyses
Bed conditioning	–	–	–	–
Bed conditioning	–	–	–	–
Bed conditioning	–	–	–	–
Loading	Every 10 BVs	Every 10 BVs	2	GEA
Feed displacement	–	Every 1 BVs	10	GEA
DI water rinse	–	Every 1 BVs	10	GEA
Elution	Every 1 BVs	–	10	GEA
Eluant rinse	Every 1 BVs	–	10	GEA
Regeneration	1 composite	–	50	ICP-AES, GEA, OH-
Composite Samples				
Effluent – 1	–	–	2	GEA
Effluent – 2	–	–	2	GEA
Effluent – 3	–	–	2	GEA
Effluent – 4	–	–	2	GEA
Effluent composite (effluents 2 - 4)	–	–	10	GEA, ICP-AES, IC, U, ICP-MS (Tc), pertechnetate
Eluate	1 composite	-	10	ICP-AES, GEA, TOC, IC, U, ICP-MS, and various radioisotopes

2.5.4 Sample Analysis

The ^{137}Cs concentration was determined using a bench-top GEA spectrometer. This allowed for rapid sample analysis. Selected sample results were later confirmed by GEA analysis in the analytical laboratory. Because of the high dose rate from ^{137}Cs , the feed sample and eluate samples required dilution before removal from the hot cell. These samples were diluted with 0.25 M NaOH and 0.5 M HNO_3 , respectively. The extent of dilution was determined by mass difference. Composite samples from each of the effluent bottles were taken for analysis by GEA. Another composite sample was taken for analysis once the contents of effluent bottles 2 through 4 were combined for Tc ion exchange. The regeneration solution was composited and a sample taken for ICP-AES and OH^- determination. Once the GEA results were confirmed for the eluate samples, the eluate samples were composited and a sample of the composite was submitted for various analyses; GEA, ICP-AES, total organic carbon (TOC), ion chromatography (IC), U, pertechnetate, and ICP-MS for ^{99}Tc .

The Na and other metal concentrations were determined with ICP-AES. The OH⁻ concentration was determined by potentiometric titration with standardized HCl. Uranium was determined using kinetic phosphorescence. The total Tc concentration was determined by ICP-MS and the pertechnetate concentration was determined using radiochemical separations specific for pertechnetate followed by beta counting. Anions were determined using IC. TOC and total inorganic carbon (TIC) were determined by silver-catalyzed hot persulfate oxidation and furnace oxidation methods.

3.0 Results and Discussion

3.1 Batch Contact Results

The K_d values were calculated using Equation 1 and are based on the ^{137}Cs concentrations as measured by GEA. The Na/Cs mole ratios were calculated based on the measured Na and total Cs concentrations in the uncontacted AP-101DF. The equilibrium Cs concentration was based on the measured ^{137}Cs concentrations and the ratio of ^{137}Cs :total Cs determined for the unspiked and spiked solutions. The Na concentration was assumed to be constant for the batch contacts. Since the quantity of H^+ added with the resin was small relative to the moles of Na^+ and OH^- in the contact solution (phase ratio of 100 mL of solution:gram of exchanger) this is a reasonable assumption. In these experiments the waste solutions were estimated to have 19.4 meq of OH^- and 49.7 meq of Na^+ , while the resin contained 0.22 meq of H^+ for the 0.1 g resin used in the batch distribution tests.⁹

The calculated ^{137}Cs K_d values are plotted in Figure 3.1 for SL-644 and IE-911 in contact with AP-101DF. The IE-911 feed condition K_d value is interpolated at 710 mL/g. The SL-644, 981112YK-N3-16/18 resin batch K_d values are defined by the following equation (linear least squares fit): $K_d = 142.44 * \ln([\text{Na}]/[\text{Cs}]) - 997.49$, $R^2 = 0.998$.

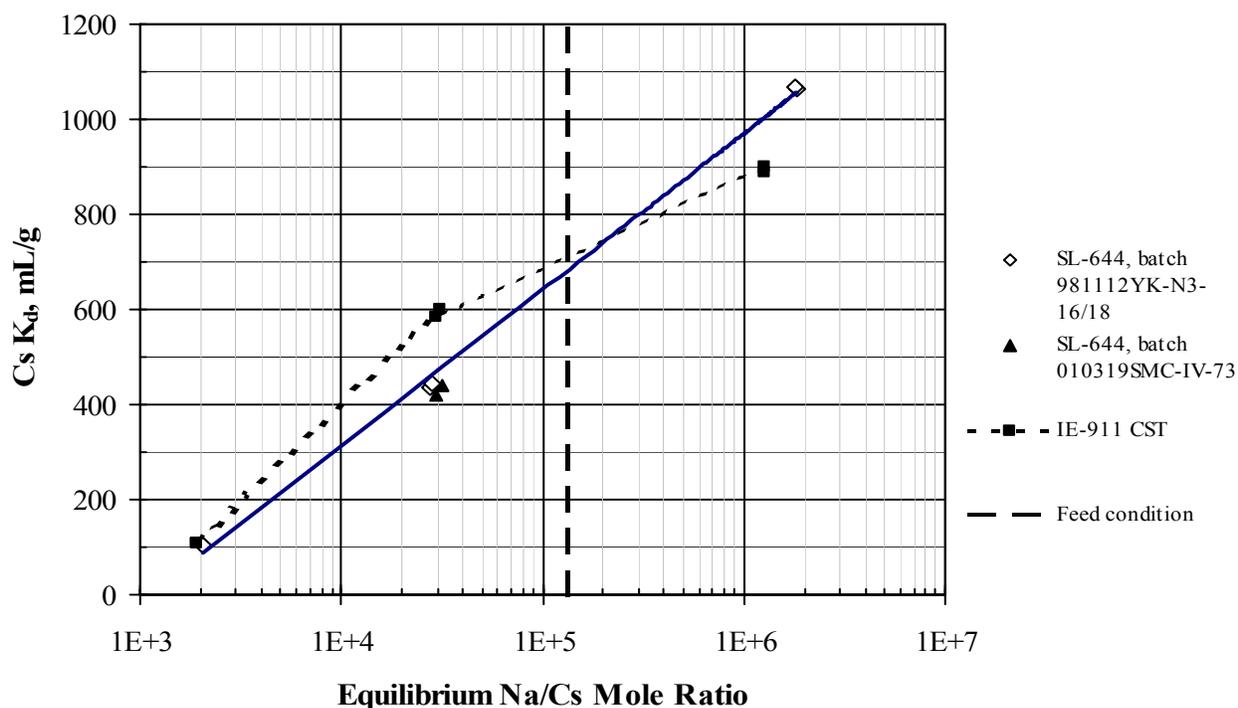


Figure 3.1. ^{137}Cs Distribution Coefficients (K_d) for AP-101DF ($T = 26^\circ\text{C}$)

⁹ The H-form resin contains 2.2 meq H^+ per gram. (Rapko, et al. 2002)

The experimental data from the single Cs concentration tested (Cs spike 3) shows the K_d value, represented by the 010319SMC-IV-73 SL-644 resin batch (212- to 425- μm particle size), is equivalent to the 981112YK-N3-16/18 material. However the slope could not be evaluated. Assuming equivalent slope, the K_d value at the AP-101DF feed condition can be estimated at 690 mL/g from the point at which the K_d curve crosses the Na/Cs mole ratio ($1.4 \text{ E}+5$) for the AP-101DF sample.

The calculated dry-bed densities determined using the resin masses and volumes in the ion exchange column system are given in Table 3.1. Good agreement was obtained between the lead and lag columns. The 0.25 M NaOH dry-bed density was equivalent to the 0.5 M HNO_3 form dry-bed density. In this case, the decrease in mass associated with the change from the Na-form to the H-form was equivalent to the decrease in volume upon contracting from the Na-form to the H-form.

Table 3.1. Dry Bed Density

Property	010319SMC-IV-73, Lead Column	010319SMC-IV-73, Lag Column
Column resin mass, ⁽¹⁾ g	4.17	4.20
Corrected column Na-form resin mass, g	2.46	2.48
Bed volume, 0.25 M NaOH, mL	10.7	10.5
Bed volume, AP-101DF, mL	9.7	9.7
Bed volume, 0.5 M HNO_3 , mL	7.7	not performed
Dry bed density, ρ, in given matrix		
Na-form resin		
0.25 M NaOH, g/mL	0.230	0.236
AP-101DF, g/mL	0.254	0.255
H-form resin		
0.5 M HNO_3 , g/mL	0.256	not performed
(1) The 212- to 425- μm particle-size resin mass measured in the as-received form.		

The calculated Cs λ values, as a function of Na/Cs mole ratio, are shown in Figure 3.2. Two Cs λ curves are plotted for SL-644. One curve represents the measured K_d values from SL-644 batch 981112YK-N3-16/18 multiplied by the calculated resin bed density of 0.15 g/mL (Fiskum, Blanchard, and Arm 2002). The other SL-644 Cs λ curve is estimated for the 010319SMC-IV-73 resin, applying the resin bed density of 0.23 g/mL to the K_d values generated for the 981112YK-N3-16/18 resin. The Cs λ value is estimated to be 170 BVs in the AP-101DF feed condition, and 160 BVs in the 0.25 M NaOH regeneration condition for 010319SMC-IV-73 SL-644. This value is the approximate point at which the Cs breakthrough curve was predicted to pass through $C/C_o = 50\%$ if sufficient feed was available for processing.

The calculated Cs λ values for IE-911 are also shown in Figure 3.2. The IE-911 bed density was determined to be 1.04 g/mL. The feed condition Cs λ value for IE-911 is estimated to be 740 BVs.

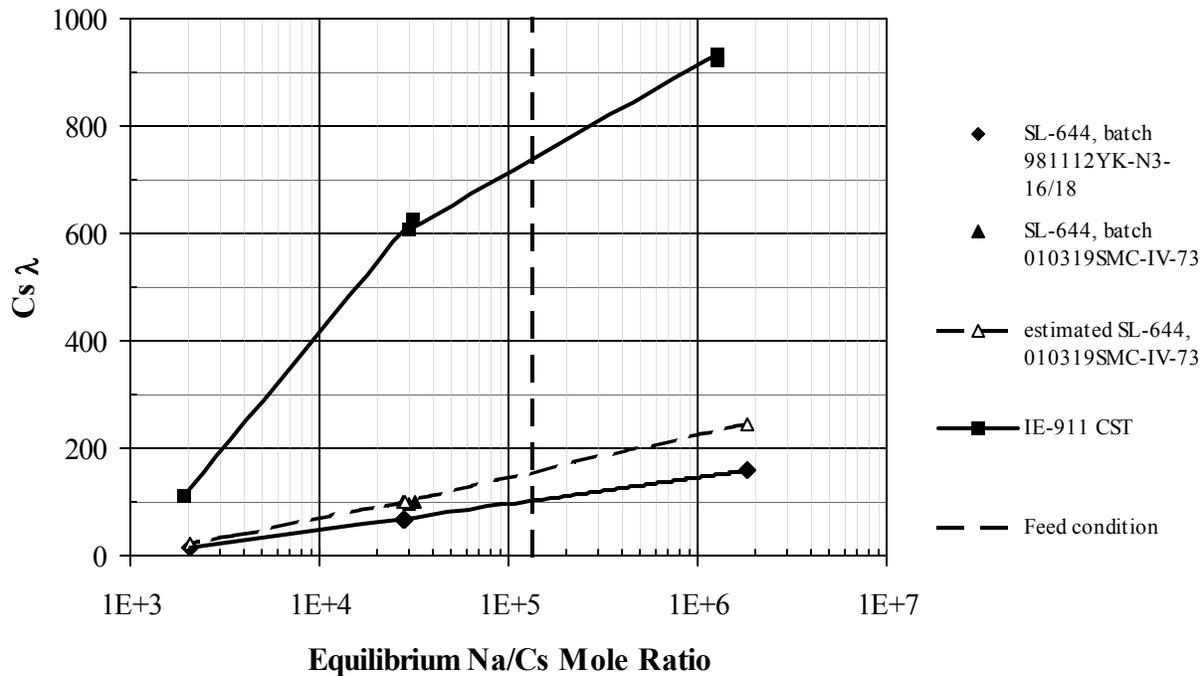


Figure 3.2. Estimated ^{137}Cs λ Values for AP-101DF (T = 26°C)

3.2 Column Test

3.2.1 Initial Resin Conditioning

The resin bed conversion progress from Na-form to H-form and vice versa could be monitored visually according to the color of the resin bed. The Na-form appeared black in the columns whereas the H-form appeared brown. Initial conversion to the Na-form in the sequential column format required nominally 104 mL (9.8 BVs fully expanded) or 2.5 AVs. During this time the BVs increased from 7.5 mL to 11 mL. Conversion to the H-form required 82 mL of 0.5 M HNO_3 (7.8 BV fully expanded) or 2.0 AVs. During this time the resin bed shrank from 11 mL to 7.8 mL. The second regeneration was performed at half the flow rate (1 BV/h) of the first regeneration. The same volume of 0.25 M NaOH was required to complete the conversion, as evidenced by visual examination.

3.2.2 Loading

After the resin beds were conditioned, the loading phase was initiated with AP-101DF. Approximately 1.5 AVs (64 mL) of effluent were initially collected in a separate collection bottle. Most of this effluent was 0.25 M NaOH from the regeneration step mixed with some AP-101DF in the ion exchange apparatus. This solution was maintained separately and not mixed with the final Cs-decontaminated AP-101DF effluent composite; thus most of the apparatus 0.25 M NaOH solution was

prevented from mixing with the AP-101DF effluent. The resin shrank about 8.5% to 9.7 mL in the AP-101DF feed.

The Cs effluent concentrations from the columns are shown in Figure 3.3 as % C/C₀ vs. the BVs of feed processed through each column. The abscissa reflects BVs as a function of the resin in the expanded regeneration condition of 10.6 mL. The C₀ value for ¹³⁷Cs was determined to be 126 μCi/mL. The C/C₀ is plotted on a probability scale as this scale tends to provide a straight-line breakthrough curve. The C/C₀ values, determined using a bench-top GEA spectrometer, were generally in good agreement with selected samples independently analyzed by the analytical laboratory. Raw analytical results and calculations are located in the appendix.

The contract ¹³⁷Cs removal limit is also shown in Figure 3.3. The C/C₀ value of 0.099% corresponds to the contract limit of 0.3 Ci/m³ for ¹³⁷Cs in the LAW glass. The C/C₀ value corresponding to this limit is determined using the Na concentration of 5.0 M in the AP-101DF, a ¹³⁷Cs feed concentration of 126 μCi/mL, a 14 wt% waste Na₂O loading in the glass, and a glass product density of 2.66 g/mL.¹⁰

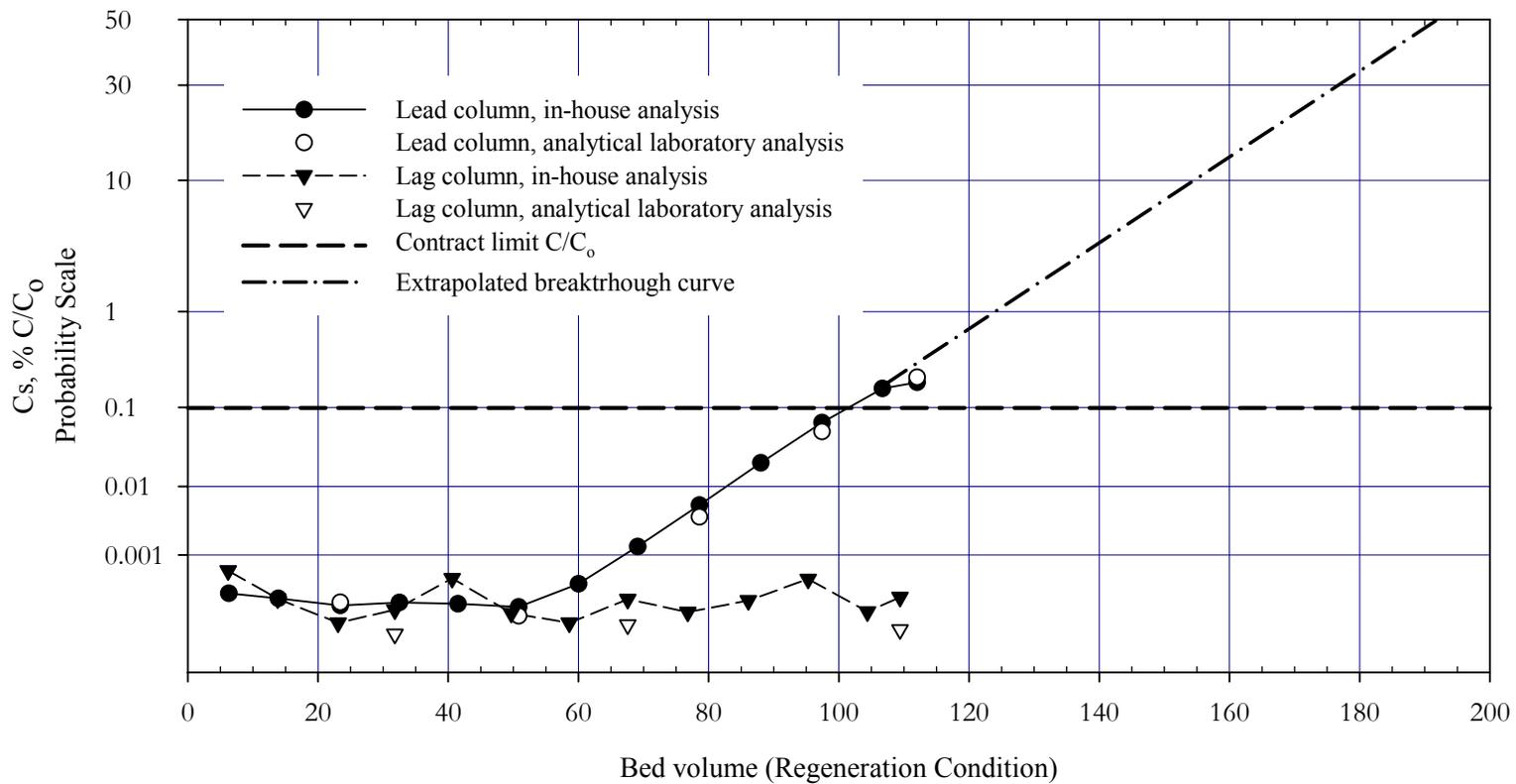
The 50% Cs breakthrough value is the point at which the C/C₀ is 50% (0.5) and is normally a direct indicator of the effective capacity of the resin. A straight-line extrapolation to 50% breakthrough from 0.2% C/C₀ is inherently prone to error. With this caveat, the 50% Cs breakthrough extrapolated to about 190 BVs (0.25 M NaOH condition) for the lead column. This value is in good agreement with the predicted Cs λ value of 160 BVs determined with batch-contact tests. There is insufficient data for an estimate of the 50% Cs breakthrough for the lag column.

The decontamination factors (DFs) were calculated on composites from each of the three effluent collection bottles and are summarized in Table 3.2. These may be compared to the contractual limit of C/C₀ = 0.099% (DF= 1,010). In all cases, sufficient Cs was removed that the contract limit for Cs removal was met. The final lag column sample C/C₀ and DF are also shown. The DF result is equivalent to the maximum DF within experimental error.

Table 3.2. Decontamination Factors for ¹³⁷Cs from AP-101DF

Sample	Volume, BV (mL)	¹³⁷ Cs Concentration μCi/mL ⁽¹⁾	C/C ₀ , %	DF
Final lag column sample	109 (1160)	7.4 E-05	7.3 E-05	1.4 E+06
Effluent composite bottle-2	34 (356)	9.8 E-05	7.7 E-05	1.3 E+06
Effluent composite bottle-3	36 (377)	1.1 E-04	8.6 E-05	1.2 E+06
Effluent composite bottle-4	40 (424)	2.3 E-04	1.8 E-04	5.5 E+05
Composite effluent	104 (1103)	1.08 E-04	8.6 E-05	1.2 E +06
(1) The ¹³⁷ Cs uncertainty ranges from 3% to 8% relative error, 1-σ.				

¹⁰ Applying a glass loading of 20 wt% would reduce the contract limit % C/C₀ to 0.069 %.



Conditions: SL-644 batch number 010319SMC-IV-73 212- to 425- μm dry particle size
 Process temperature = 28 $^{\circ}\text{C}$
 BV in 0.25 M NaOH feed condition = 10.6 mL
 Flow Rate = 2.8 BV/h
 ^{137}Cs $C_0 = 126 \mu\text{Ci/mL}$
 Na concentration = 4.97 M.

Figure 3.3. ^{137}Cs Breakthrough Curves for AP-101DF Sample, Probability Plot

The three composite effluent fractions were combined into a single composite for subsequent processing. This Cs-decontaminated effluent was characterized and the results are summarized in Table 3.3. Major constituents (Na, K, Al, NO₃, and NO₂) in the effluent were similar in concentration to the feed within experimental error. The authors have no explanation for the ~25% increase measured for total Tc and OH other than experimental/analytical error. The pertechnetate remained constant within experimental error. Thus the total Tc determined by ICP-MS may be biased high in this analysis and/or biased low in the feed analysis.¹¹ Notably nearly all U and Pu¹² were removed from the AP-101DF by SL-644. The F, PO₄ and SO₄ also resulted in low effluent recovery. The low recovery is thought to be due to analytical error/uncertainty.

3.2.3 Feed Displacement and Rinse

There was a delay between the introduction of the feed displacement and the collection of these samples from the effluent line. Approximately 42 ml of Cs-decontaminated AP-101DF were collected as effluent prior to starting the feed displacement sampling. The relative Cs concentration, as C/C_o , was $< 4 \text{ E-}3 \%$ for all feed displacement and DI water rinse samples.

3.2.4 Elution and Eluant Rinse

The elution proceeded similarly as found in the simulant test with respect to the Cs elution profile and resin bed shrinkage. However, the characteristic color change from black in the Na-form to tan in the H-form was not visible. The resin appeared virtually black for the entire elution and rinse cycles.

The lead column C/C_o ¹³ values for ¹³⁷Cs are shown in Figure 3.4 for the elution and the eluant rinse steps. The ordinate is a logarithmic scale to clearly show the large range of C/C_o values obtained. The abscissa is given in BVs relative to the regeneration condition. The majority of the ¹³⁷Cs was contained in elution BVs 3-5. The peak value of C/C_o was found to be 64. The elution cutoff of $C/C_o = 0.01$ was reached at 12 BVs but elution was continued beyond this due to the lag between sample collection and the determination of the Cs concentration. At 18.5 BVs the C/C_o visibly rises. At this point, the columns were allowed to sit in 0.5 M HNO₃ for about two hours while samples were counted. Cesium continued to elute from the resin and when flow was re-started, showed a slight rise in relative Cs concentration. The C/C_o values for the eluant rinse with DI water dropped rapidly in ¹³⁷Cs concentration indicating DI water did not continue Cs elution.

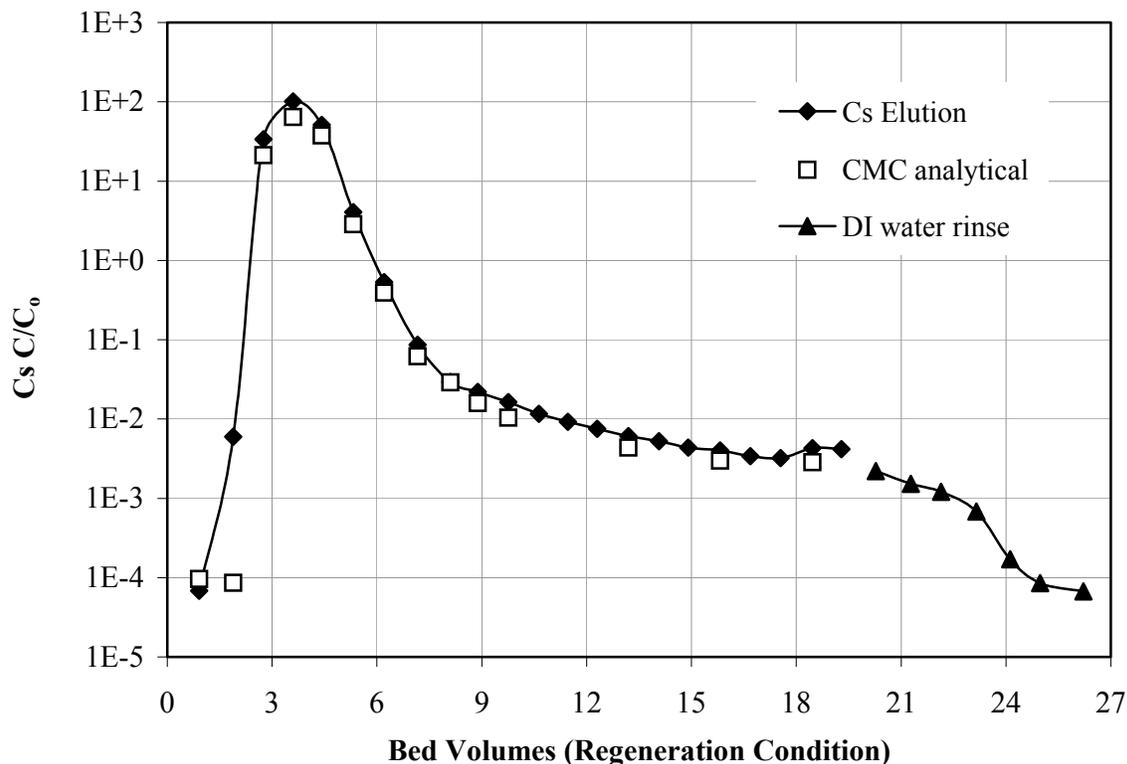
¹¹ Fiskum et al. 2000 reported undiluted AP-101 tank waste total ⁹⁹Tc concentration of 4.90E-2 μCi/mL. Applying the dilution factor of 1.13 to this value results in a calculated ⁹⁹Tc concentration of 4.34E-2 μCi/mL in the AP-101DF. The measured feed value of 3.93E-2 μCi/mL is 10% lower than the calculated value, indicating a possible slight low bias in the measured feed concentration.

¹² Pu concentration in the effluent was estimated from the Pu concentration in the Tc ion exchange effluent. This assumes that the Tc ion exchange processing did not remove Pu.

¹³ The C_o refers to the ¹³⁷Cs concentration in the AP-101 DF sample fed to the lead column. For elution, the C/C_o value is an indication of the extent to which ¹³⁷Cs is concentrated relative to the feed. It is an indirect measure of the extent to which the resin is actually eluted.

Table 3.3. AP-101DF Cs-Decontaminated Product Effluent

Component	Concentration, M	% Change from Feed
Cations		
Na ⁺	4.81 E+0	-3.0
K ⁺	7.44 E-1	-2.2
Uranyl	3.8 E-6	-98
Anions		
AlO ₂ ⁻	2.45 E-1	-5.3
F ⁻	9.74 E-2	-19.
Cl ⁻	4.09 E-2	-1.2
CrO ₄ ⁻²	2.70 E-3	-7.5
NO ₂ ⁻	7.78 E-1	10.
NO ₃ ⁻	1.88 E+0	12.
OH ⁻	2.40 E+0	24
PO ₄ ⁻³ (IC)	7.3 E-3	-27.
P (ICP-AES)	1.06 E-2	-15.
SO ₄ ⁻²	3.25 E-2	-33.
Oxalate	1.1 E-2	-2.7
Radionuclides	μCi/mL	% Change from Feed
⁶⁰ Co	2.23 E-3	-12.
⁹⁹ Tc	4.99 E-02	27.
⁹⁹ Tc as pertechnetate	3.95 E-2	14.
¹³⁷ Cs	1.08 E-4	>-99
¹⁵⁴ Eu	< 3.0 E-5	NA
²³⁹⁺²⁴⁰ Pu	1.74E-5 (estimated) ⁽¹⁾	-84 ⁽¹⁾
Solution density, g/mL	1.257	0.08
NA = not applicable, not detected in feed or effluent		
(1) Pu removal estimated based on feed Pu concentration and effluent Pu concentration from Tc ion exchange. The ²³⁹⁺²⁴⁰ Pu concentration in the Tc-removed AP-101DF effluent was 1.74E-5 μCi/mL. The Pu removal estimate is supported by the eluate analysis where 72% Pu was recovered.		



Conditions: SL-644 batch number 010319SMC-IV-73 212- to 425- μ m dry particle size
 Process temperature = 28-29 °C
 BV in 0.25 M NaOH feed condition = 10.6 mL
 Flow Rate = 0.88 BV/h
 ^{137}Cs C_0 = 126 $\mu\text{Ci/mL}$
 Eluant is 0.5 M HNO_3 .

Figure 3.4. ^{137}Cs Elution and Eluant Rinse of the Lead Column

The eluate samples from the lead column were composited and a sub-sample taken for analysis. The remainder of the composite was saved for vitrification testing. The analytical results are shown in Tables 3.4 and 3.5. Sodium was the dominant component detected using ICP-AES with some Ca, Cd, Cr, Fe, K, Pb, U, and Pu eluting as well. The relative fractions of these analytes recovered in the eluate are given in the tables. The U recovered in the eluate (~42 mg) represents 87% of the U loaded in the AP-101DF feed (48 mg). Plutonium (based on $^{239+240}\text{Pu}$) is also largely recovered (72%) in the eluate.¹⁴ A significant fraction (57%) of the Cm was also recovered in the eluate. The other analytes recovered to a much

¹⁴ The sum of ^{239}Pu and ^{240}Pu measured by ICP-MS (8.98E-4 $\mu\text{Ci/mL}$) was a factor of two higher than the radiochemistry determination of the $^{239+240}\text{Pu}$ (4.63E-4 $\mu\text{Ci/mL}$). The ICP-MS determination is considered biased high.

smaller percentage in the eluate (Na: 0.14%; Ca: 2%; Cr: 1%; Fe: 29%; K: 0.03%; Pb: Sr: 7%). Because the feed concentrations were “less than” values, the Cd and Pb recovered at >10% and >8%, respectively. The B and Si were probably leached from the glass vials used during the sample preparation process; the preparative blank sample was nearly as high in B and Si concentrations as the eluate sample. Thus the reported B and Si concentrations are not indicative of the eluate composition. As expected ¹³⁷Cs was the dominant radionuclide detected. The only anion detected was nitrate, which is not surprising since the eluant was 0.5 M HNO₃.

Most of the specified minimum reportable quantity (MRQ) levels were met with some exceptions. The large amount of ¹³⁷Cs prevented the detection limits for ²⁴¹Am, ¹⁵⁴Eu, and ¹⁵⁵Eu from meeting the MRQ levels. Relatively high ¹³⁷Cs activity increased the gamma background level in the detectors due to Compton scattering, thereby making it difficult to detect lower concentrations of other gamma emitters. The large amount of NO₃ prevented the detection limit for Cl from meeting the MRQ level of 3 µg/mL. The large NO₃ concentration required large sample dilutions and increased the method detection limit for the other components. The anion concentrations other than nitrate are expected to be small. The TIC analysis was not completed because carbonate is known to evolve as CO₂ in acidic solutions.

Table 3.4. Inorganic and Organic Analytes in the Lead Column Eluate Composite

Analyte	Method	MRQ µg/mL	Cs eluate µg/mL	% recovered	Analyte	Method	MRQ µg/mL	Cs eluate µg/mL	% recovered
Ag	ICP-AES	NMRQ	< 0.63	ND	Rh	ICP-MS	NMRQ	5.7 E-2	NM
Al	ICP-AES	75	18.6	0.05	Ru	ICP-MS	NMRQ	3.2 E-3	NM
As	ICP-AES	NMRQ	< 6.3	ND	Sb	ICP-AES	NMRQ	< 13	ND
B	ICP-AES	NMRQ	194	HB	Se	ICP-AES	NMRQ	< 6.3	ND
Ba	ICP-AES	2.3	[0.29]	[>4] ⁽¹⁾	Si	ICP-AES	170	145	HB
Be	ICP-AES	NMRQ	< 0.25	ND	Sn	ICP-AES	1500	< 38	ND
Bi	ICP-AES	NMRQ	< 2.5	ND	Sr	ICP-AES	NMRQ	< 0.38	ND
Ca	ICP-AES	150	[25]	[2.0]	Ta	ICP-MS	NMRQ	2.7E-3	NM
Cd	ICP-AES	8	[1.2]	[>10] ⁽¹⁾	Te	ICP-AES	NMRQ	< 38	ND
Ce	ICP-AES	NMRQ	< 5.0	ND	Th	ICP-AES	NMRQ	< 25	ND
Co	ICP-AES	30	< 1.3	ND	Ti	ICP-AES	17	< 0.63	ND
Cr	ICP-AES	15	12	1.4	U	ICP-AES	600	[160]	[67]
Cs ⁽²⁾	GEA	1.5	37	130 ⁽³⁾	U	KPA	600	206 ⁽⁴⁾	87
Cu	ICP-AES	17	[1.2]	[> 6] ⁽¹⁾	V	ICP-AES	NMRQ	< 1.3	ND
Dy	ICP-AES	NMRQ	< 1.3	ND	Y	ICP-AES	NMRQ	< 1.3	ND
Eu	ICP-AES	NMRQ	< 2.5	ND	Zn	ICP-AES	17	< 1.3	< 2.8
Fe	ICP-AES	150	[6.1]	[29]	Zr	ICP-AES	NMRQ	< 1.3	ND
K	ICP-AES	75	[57]	[0.03]	TOC	Hot Pers.	1500	< 30	< 0.3
La	ICP-AES	35	< 1.3	ND	TOC	Furnace	1500	180	0.4

Analyte	Method	MRQ µg/mL	Cs eluate µg/mL	% recovered	Analyte	Method	MRQ µg/mL	Cs eluate µg/mL	% recovered
Li	ICP-AES	NMRQ	< 0.75	ND	Br ⁻	IC	NMRQ	< 63	ND
Mg	ICP-AES	300	< 2.5	ND	Cl ⁻	IC	3	< 63	< 0.7
Mn	ICP-AES	150	< 1.3	ND	F ⁻	IC	150	< 63	< 0.5
Mo	ICP-AES	150	< 1.3	ND	NO ₂ ⁻	IC	3000	< 125	< 0.1
Na	ICP-AES	75	912	0.14	NO ₃ ⁻	IC	3000	29250	NA
Nd	ICP-AES	NMRQ	< 2.5	ND	PO ₄ ⁻³	IC	2500	< 125	ND
Ni	ICP-AES	30	[1.6]	[6]	SO ₄ ⁻²	IC	2300	< 125	< 0.5
P	ICP-AES	600	< 2.5	< 0.1	Oxalate	IC	NMRQ	< 125	ND
Pb	ICP-AES	300	[4.7]	[> 6] ⁽¹⁾	OH	Titration	17	NM	NM
Pd	ICP-MS	NMRQ	5.9 E-3	NM	Wt% dried solids		0.1	0.34%	NA
Pr	ICP-MS	NMRQ	5.2 E-4	NM	Wt% oxides		NMRQ	0.40%	NA
Pt	ICP-MS	NMRQ	3.0E-2	NM	Density, g/mL		NMRQ	1.011	NA

ASR 6192, sample ID 01-1842

Notes: MRQ is minimum reportable quantity requested by Bechtel. NMRQ is no minimum reportable quantity requested. The overall error is estimated to be within +/-15%. Values in brackets are within 10-times the detection limit and errors are likely to exceed +/-15%.

NA = not applicable

ND = analyte not detected in the feed or product

NM = analyte not measured in the eluate

HB = high preparative blank concentration, recovery cannot be calculated

(1) Analyte was not detected in the feed.

(2) Calculated based on ¹³⁷Cs concentration in the eluate and applied Cs isotopic distribution ratio (¹³³Cs 60.6 wt%, ¹³⁵Cs 15.4 wt%, ¹³⁷Cs 24.0 wt%).

(3) Calculated based on Cs feed concentration of 4.89 µg/mL. If the feed Cs concentration is actually 6.04 µg/mL, then Cs recovery is calculated to be 105%. See footnote (1) of Table 2.3.

(4) Total U measured by ICP-MS was in good agreement at 190 µg/mL.

Table 3.5. Radionuclides in the Lead Column Eluate Composite⁽¹⁾

Analyte	Method	MRQ ⁽²⁾ μCi/mL	Cs eluate μCi/mL	Error %	% recovered	Analyte	Method	MRQ ⁽²⁾ μCi/mL	Cs eluate μCi/mL	Error %	% recovered
³ H	Radchem	NMRQ	1.72 E-4	10	NM	¹⁵⁵ Eu	GEA	9.00 E-2	<3 E-1	—	ND
¹⁴ C	Radchem	NMRQ	2.4 E-5	30	NM	²³² Th	GEA	NMRQ	<2 E-1	—	NM
⁵¹ Cr	GEA	NMRQ	< 2 E-2	—	NM	²³³ U	ICP-MS	NMRQ	1.78 E-4	1	NM
⁵⁴ Mn	GEA	NMRQ	<3 E-2	—	NM	²³⁴ U	ICP-MS	NMRQ	9.89 E-5	4	NM
⁵⁹ Fe	GEA	NMRQ	<3 E-2	—	NM	²³⁵ U	ICP-MS	NMRQ	3.53 E-6	1	NM
⁶⁰ Co	GEA	NMRQ	<5 E-3	—	<34	²³⁶ U	ICP-MS	NMRQ	7.17 E-6	2	NM
⁶³ Ni	Radchem	NMRQ	8.83 E-4	5	NM	²³⁸ U	ICP-MS	NMRQ	6.31 E-5	0.5	NM
⁷⁹ Se	Radchem	NMRQ	3.52 E-6	25	NM	²³⁶ Pu	Radchem	NMRQ	< 4 E-7	—	NM
⁸⁸ Y	GEA	NMRQ	<2 E-2	—	NM	²³⁷ Np	ICP-MS	NMRQ	6.25 E-7	13	ND
⁹⁰ Sr	Radchem	1.50 E-1	2.95 E-2	3	7	²³⁸ Pu	Radchem	NMRQ	6.00 E-5	4	74
⁹⁵ Nb	GEA	NMRQ	<2 E-2	—	NM	²³⁹⁺²⁴⁰ Pu	Radchem	NMRQ	4.63 E-4	2	72
⁹⁹ Tc	ICP-MS	3.00 E-3	1.36 E-4	10	0.06	²³⁹ Pu	ICP-MS	NMRQ	7.64 E-4	3	NM
¹⁰³ Ru	GEA	NMRQ	<2 E-1	—	NM	²⁴⁰ Pu	ICP-MS	NMRQ	1.34 E-4	13	NM
¹⁰⁶ Ru	GEA	NMRQ	<7 E-1	—	NM	²⁴¹ Pu	Radchem	NMRQ	3.06 E-3	8	NM
¹¹³ Sn	GEA	NMRQ	<2 E-1	—	NM	²⁴¹ Am	Radchem	7.20E-04	3.04 E-5	3	3.8
¹²⁵ Sb	GEA	NMRQ	<4 E-1	—	NM	²⁴¹ Pu, ²⁴¹ Am	ICP-MS	NMRQ	<1.6 E-4 μg/mL ⁽⁵⁾	—	NM
¹²⁶ Sn/Sb	GEA	NMRQ	2.69 E-1	19	NM	²⁴² Am	Radchem	NMRQ	< 3 E-7	—	NM
¹²⁹ I	ICP-MS	NMRQ	< 6 E-6	—	NM	²⁴² Cm	Radchem	NMRQ	<2 E-7	—	ND
¹³⁴ Cs	GEA	NMRQ	1.56 E-1	6	NM	²⁴² Pu	ICP-MS	NMRQ	<1.6 E-6	—	NM
¹³⁷ Cs	GEA	5.00 E-2	7.65 E+2	2	104	²⁴³⁺²⁴⁴ Cm	Radchem	NMRQ	7.30 E-6	6	57
¹⁴⁴ Ce	GEA	NMRQ	<6 E-1	—	NM	Alpha sum ⁽⁴⁾	Radchem	NMRQ	5.60 E-4	2	36
¹⁵¹ Sm ⁽³⁾	Radchem	NMRQ	2.42 E-4	6	NM	Total alpha	Radchem	2.30E-01	9.13 E-4	11	
¹⁵² Eu	GEA	NMRQ	<1 E-2	—	NM	Total beta	Radchem	NMRQ	7.97 E+2	4	NM

Analyte	Method	MRQ ⁽²⁾ μCi/mL	Cs eluate μCi/mL	Error %	% recovered	Analyte	Method	MRQ ⁽²⁾ μCi/mL	Cs eluate μCi/mL	Error %	% recovered
¹⁵⁴ Eu	GEA	2.00 E-3	<3 E-2	—	ND						
Notes: NM = not measured in the feed ND = not detected in feed or eluate (1) ASR 6192, Sample 01-1842 (2) MRQ is minimum reportable quantity requested by Bechtel. NMRQ is no minimum reportable quantity requested. (3) The ¹⁵¹ Sm is an upper bound because other beta-emitters interfered with the analysis. (4) The alpha sum (total of individually-measured Pu, Am, and Cm alpha emitters) provides the best estimate of the total alpha activity in the sample. (5) The AMU-241 is reported in units of μg/mL. Applying the specific activity of ²⁴¹ Pu, sample activity is <1.6E-2 μCi/mL; applying the specific activity of ²⁴¹ Am, sample activity is <5.4E-4 μCi/mL.											

3.2.5 Regeneration

The lead column was regenerated with 50 mL 0.25 M NaOH, collected in 1 composite fraction. The composition of this regeneration effluent solution is shown in Table 3.6. Sodium was the only cation found above the blank concentration. The charge balance between Na and OH are not equal. The counter-ion for the extra Na is predicted to be residual NO₃ from the elution step.

Table 3.6. Composition of Regeneration Solution

Analyte	Concentration, µg/mL	Concentration, M
Na ⁺	1020	0.044
K ⁺	< 40	< 1E-3
OH ⁻	500	0.030
Total Cs ⁽¹⁾	2.8 E-3	2.1 E-8
¹³⁷ Cs	6.69 E-4	5.85 E-2 µCi/mL
Density, g/mL	1.006	
(1) Total Cs is calculated from the isotopic ratio and measured ¹³⁷ Cs concentration		

3.2.6 Activity Balance for ¹³⁷Cs

An activity balance for ¹³⁷Cs was completed to compare the ¹³⁷Cs recovered in various process streams to the ¹³⁷Cs present in the feed sample (Table 3.7). As expected, virtually all ¹³⁷Cs was found in the eluate, recovering >99 % of the ¹³⁷Cs present in the initial AP-101DF feed. The lag column remained loaded with 35 µCi ¹³⁷Cs based on the integration of the lead column breakthrough curve.

Table 3.7. Activity Balance for ¹³⁷Cs

Solution	¹³⁷ Cs, µCi	¹³⁷ Cs in Feed Sample, %
Feed Sample	1.50 E+5	1.00 E+2
Effluent	1.3 E-1	8.4 E-5
Load samples	1.2 E+0	8.2 E-4
Feed displacement	2.6 E-1	1.7 E-4
DI Water Rinse	2.1 E-1	1.4 E-4
Column #1 Eluate	1.56 E+5	1.04 E+2
Column #1 DI water rinse	7.6 E+0	5.1 E-3
Column #1 regeneration	3.0 E+0	2.0 E-3
Lag column Cs loading	3.5 E+1	2.3 E-2
Total ¹³⁷ Cs Recovery	1.56 E+5	1.04 E+2

3.2.7 SL-644 Resin Volume Changes

The SL-644 resin is known to change in volume as a function of the solution pH and ionic strength (Hassan et al., 1999). The resin BV change history is shown in Table 3.8. The volume contraction after each subsequent 0.5 M HNO₃ step became more pronounced with cycling. The first volume contraction stabilized at 9.2 mL; the final measured volume contraction stabilized at 7.7 mL. The variation in BV as a function of the process steps for both columns is shown in Figure 3.5 a and b. In Figure 3.5a, the BVs are normalized to the volume in the 0.25 M NaOH regeneration condition just prior to AP-101DF loading. Two process cycles are shown with the first cycle consisting of the bed conditioning steps and the second cycle consisting of the actual process test. Each process step is denoted with a number given in Table 3.8. In Figure 3.5b, the observed volume changes show clearly the greater contraction and slightly reduced expansion. Fluidizing the bed in the H-form resulted in tighter resin packing, yet the subsequent expansion in the Na-form appears to have been unaffected in that the regeneration volume is equivalent to that obtained prior to fluidization. Overall, there appears to be a general decline in resin volume in both the H-form and the Na-form. The change in resin volume may be caused in part by small losses of the resin during processing. Resin volume changes may also be attributed to compaction while swelling during the regeneration step.

Table 3.8. SL-644 Bed Volume Changes

Feed	Symbol	Process Step	BV 010319SMC-IV-73 212-425 μm PSD	
			Lead column, mL ⁽¹⁾	Lag column, mL ⁽²⁾
Initial packing	P	1	10.9	10.9
0.5 M HNO ₃	E	2	9.2	8.9
DI water	W	3	9.2	8.9
0.25 M NaOH	R	4	11.2	10.8
AW-101 simulant	F	5	10.2	10.0
0.1 M NaOH	FD	6	10.9	10.7
DI water	W	7	10.9	10.9
0.5 M HNO ₃	E	8	8.9	8.9
DI water	W	9	8.9	8.7
Re-fluidize bed	RP	10	7.5	7.5
0.25 M NaOH	R	11	11.0	10.9
DI water	W	12	10.5	11.0
0.5 M HNO ₃	E	13	7.9	7.7
DI water	W	14	7.9	7.7
0.25 M NaOH	R	15	10.7	10.5
AP-101DF	F	16	9.7	9.7
0.1 M NaOH	FD	17	10.5	10.4
DI water	W	18	10.4	10.5
0.5 M HNO ₃	E	19	7.7	—
DI water	W	20	7.7	—
0.25 M NaOH	R	21	10.5	—
DI water	W	22	10.2	—

(1) Calculated resin mass after washing, dry Na-form: 2.46 g.
(2) Calculated resin mass after washing, dry Na-form: 2.48 g.
Note: The inside diameter of each column was 1.46 cm.

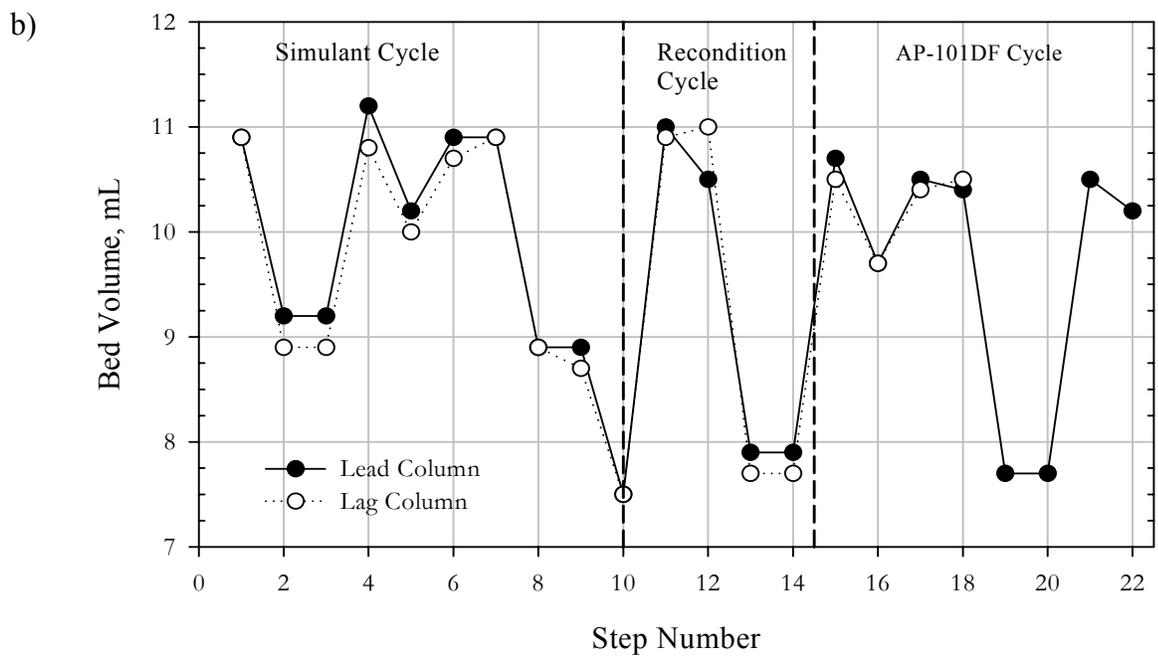
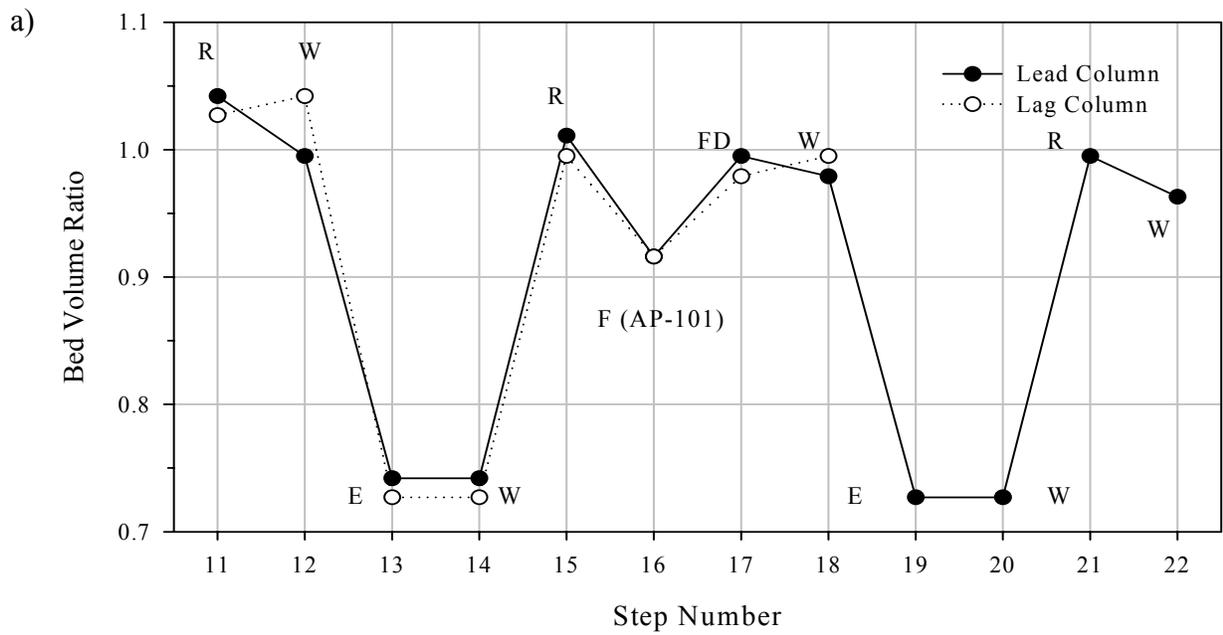


Figure 3.5. (a and b) Comparison of Bed Volumes of the Lead and Lag Columns (SL-644 Batch 010319SMC-IV-73, 212- to 425- μm)
 a) Relative BV for reported testing cycle; baseline BV = 10.6 mL in the 0.25 M NaOH Regeneration Condition.
 b) Actual BVs for all tested cycles, including initial simulant run.

4.0 Conclusions

The objectives of the testing were met.

Batch distribution values were developed as a function of Na/Cs mole ratios for both SL-644 and IE-911 in AP-101DF.

- The SL-644 equilibrium data resulted in a K_d value of 690 mL/g, corresponding to a predicted Cs λ of 160 BVs (0.25 M NaOH condition), at a Na/Cs mole ratio of $1.4 \text{ E}+5$, 25°C.
- The IE-911 CST equilibrium data resulted in a K_d value of 710 mL/g, corresponding to a Cs λ value of 740 BVs, at a Na/Cs mole ratio of $1.4\text{E}+5$, 25 °C.

Cs decontamination from AP-101DF (Envelope A) was successfully demonstrated.

- An overall DF of $7.8 \text{ E}+5$ was obtained after processing 112 BVs providing a Cs-decontaminated effluent with a ^{137}Cs concentration of $1.1 \text{ E}-4 \text{ }\mu\text{Ci/mL}$. This represents 0.09% of the contract limit of $1.25 \text{ E}-1 \text{ }\mu\text{Ci/mL}$ in the treated effluent (based on 14 wt% waste Na_2O loading).¹⁵

Cs load and elution breakthrough profiles were developed.

- The estimated 50% Cs breakthrough for the lead ion exchange column, extrapolated from 0.2% C/C_0 to 50% C/C_0 , was about 190 BVs (0.25 M NaOH regeneration condition). This is a huge extrapolation and as such is inherently uncertain, however it is nearly equivalent to the predicted 160 BVs based on batch-contact studies.
- The Cs-loaded lead column was efficiently eluted with 0.5 M HNO_3 . Over 94% of the ^{137}Cs was eluted from the column in 2.5 BVs of eluate. A total of 12 BVs eluate were required to reach the elution end point of $C/C_0 = 0.01$. The peak C/C_0 value for ^{137}Cs was 64 for the lead column. The activity balance accounted for 104% of the feed ^{137}Cs , which is essentially equivalent to 100% within experimental error.

The Cs eluate solution was composited and characterized in preparation for subsequent evaporation and HLW vitrification testing.

- A high percentage of the U (87%) and Pu (72%) was exchanged onto the resin and recovered in the acid eluate.

¹⁵ The effluent ^{137}Cs concentration was 0.13% of the contract limit of $0.0874 \text{ }\mu\text{Ci/mL}$ in the treated effluent based on the maximum waste loading of 20 wt% waste Na_2O .

The effectiveness of all SL-644 ion exchange process steps was demonstrated.

- The SL-644 was adequately regenerated with 4.7 BVs or 2.2 AVs of 0.25 M NaOH. The average ^{137}Cs concentration in the regeneration solution was $5.8 \text{ E-}2 \text{ } \mu\text{Ci/mL}$, equivalent to C/C_0 of $4.6 \text{ E-}4$.
- An activity balance for ^{137}Cs indicated 104% of the ^{137}Cs present in the feed sample was accounted for in the samples and process streams (mostly in the eluate) indicative of good experimental integrity.
- No fouling, or other off-normal conditions, of the resin bed was observed.

5.0 References

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Appendix A

Calculations

Cs-137 Contractual Limit in AP-101 Env. A Vit feed

Assumptions

- 1) Concentration of Na₂O in Env. A glass = 14% (=14 g Na₂O / 100 g glass)
- 2) For maximum ¹³⁷Cs concentration in glass assume all Na comes from the feed. If some Na is added to Vit feed, multiply the maximum ¹³⁷Cs value determined below by ratio of total Na:feed Na.
- 3) Glass density = 2.66 MT/m³ (=2.66 g/mL)
- 4) Maximum Cs-137 in glass = 0.3 Ci/m³ (= 0.3 Ci / 1E+6 mL = 3E-7 Ci/mL)
- 5) AP-101DF actual waste and AW-101 simulant Na concentration = 5 M
- 6) AP-101DF actual waste ¹³⁷Cs concentration = 126 μCi / mL

Na Loading in Glass

$$14 \text{ g Na}_2\text{O} / 100\text{g glass} * 1 \text{ mole Na}_2\text{O} / 62 \text{ g Na}_2\text{O} * (2 \text{ mole Na} / \text{mole Na}_2\text{O}) * (23 \text{ g Na} / \text{mole Na}) * (2.66 \text{ g glass} / \text{mL glass}) = 0.276 \text{ g Na} / \text{mL glass}$$

Maximum ¹³⁷Cs:Na in glass

$$(3.0\text{E-}7 \text{ Ci } ^{137}\text{Cs} / \text{mL glass}) / (0.276 \text{ g Na} / \text{mL glass}) = 1.09 \text{ E-}6 \text{ Ci } ^{137}\text{Cs} / \text{g Na}$$

$$(1.09 \text{ E-}6 \text{ Ci } ^{137}\text{Cs} / \text{g Na}) * (23 \text{ g Na} / \text{mole}) = 2.50\text{E-}5 \text{ Ci } ^{137}\text{Cs} / \text{mole Na}$$

Maximum ¹³⁷Cs:Na in feed

$$\begin{aligned} (2.5\text{E-}5 \text{ Ci } ^{137}\text{Cs} / \text{mole Na}) * (5 \text{ mole Na} / \text{L feed}) &= 1.25 \text{ E-}4 \text{ Ci } ^{137}\text{Cs} / \text{L} \\ &= 125 \text{ } \mu\text{Ci } ^{137}\text{Cs} / \text{L} \\ &= 0.125 \text{ } \mu\text{Ci } ^{137}\text{Cs} / \text{mL} \end{aligned}$$

AP-101DF actual waste Cs fraction remaining (C/Co) Contractual Limit

$$\begin{aligned} (0.125 \text{ } \mu\text{Ci } ^{137}\text{Cs} / \text{mL}) / (126 \text{ } \mu\text{Ci } ^{137}\text{Cs} / \text{mL}) &= 9.92\text{E-}4 \text{ C/C}_o \\ &= 0.0992 \% \text{ C/C}_o \end{aligned}$$

Decontamination Factor (DF) Contract Limit

$$1 / (9.92\text{E-}4 \text{ C/C}_o) = 1008 \text{ C}_o/\text{C}$$

Appendix B

Sample Identification

Table B.1. Sample Identification

Sample Description	ASR	RPL ID	Sample Identification	Extended Sample Description
Batch Contact Testing				
First batch contact with SL-644 981112YK-N3-16/18	6048	01-0575 -- 01-0576	AP101-644a and duplicate	Batch contact with SL-644, no spike
		01-0577 -- 01-0578	AP101-S1-644a and duplicate	Batch contact with SL-644, 1E-3 M Cs spike
		01-0579 -- 01-0580	AP101-S2-644a and duplicate	Batch contact with SL-6444, 5E-3 M Cs spike
Batch contact with IE-911		01-0581 -- 01-0582	AP101-CSTa and duplicate	Batch contact with IE-911 no spike
		01-0583 -- 01-0584	AP101-S1-CSTa and duplicate	Batch contact with IE-911, 1E-3M Cs spike
		01-0585 -- 01-0586	AP101-S2-CSTa and duplicate	Batch contact with IE-911, 5E-3M Cs spike
Controls		01-0587	AP101-S1a	Batch contact control (no exchanger) 1E-3M Cs
		01-0588	AP101-S2a	Batch contact control (no exchanger) 5E-3M Cs
		01-0589	AP101-Ca	Batch contact control (no exchanger), unspiked
Second batch contact with SL-644 010319SMC-IV-73	6130	01-1336--01-1337	AP101-S3-644-F/A and duplicate	Batch contact samples with SL-644, 1E-3M Cs
		01-1338	AP101- S3C-F/A	Batch contact control (no exchanger) Spike 3
		01-1339 -- 01-1340	AP101-S2a and S1a	Re-analysis of controls, Spike 1 and Spike 2
Column Run				
Initial Feed Sample, AP-101DF	6031	01-0520	AP-101DF	Initial feed sample
Load - Elution samples	6097	01-0780	AP-101-F0	Initial feed sample
		01-0781 -- 01-0785	AP-101L-F3 through F-13	Lead column load samples
		01-0786 -- 01-0788	AP-101P-F4 through F-13	Lag column load samples
		01-0789 -- 01-0791	AP101-Fcomp2-A through Fcomp4-A	Composite effluent samples
		01-0792 -- 01-0794	AP101-FD2-A through FD6-A	Feed displacement samples
		01-0795 -- 01-0796	AP101-DI2-A and DI5-A	Water rinse samples
		01-0797 -- 01-0809	AP101L-E1-DA	Lead column elution analytical samples
		01-0810	AP101L-RGN-A	Regeneration solution analytical sample
AP-101DF effluent composite	6121	01-1203	AP1-Tc-0-C	AP-101DF Cs IX effluent composite
AP-101 Cs eluent	6192	01-1842	AP101L-E-Comp	AP-101DF Cs eluant

Notes: 1) The suffix letter “a” after the sample ID indicates the sample was loaded out of the hot cell into a clean analytical vial; the suffix letter “D” after the sample ID indicates the sample was diluted prior to submission for analysis. Unidentified samples are associated with other tests unrelated to this work.

Appendix C

AP-101DF Batch Contact Analytical Results

Appendix D

AP-101DF Column Processing Calculations and Results

Appendix E

AP-101DF Column Processing Analytical Results

Appendix F

Cesium Ion Exchange and Batch Contacts Testing Personnel

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